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ADSORBABLE ORGANIC HALIDE (AOX) SAMPLING IN THE ATHABASCA AND WAPITI-SMOKY RIVERS

FALL & WINTER 1989-90

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EXECUTIVE SUMMARY

Adsorbable organic halide (AOX) has become an accepted measure of chlorinated organic material, and is used to monitor and regulate bleached kraft pulp mill effluents (BKME). Two surveys on the Wapiti-Smoky river system and one on the Athabasca River were carried out in 1989-90 to assess the presence and downstream persistence of AOX. Both of these river systems receive BKME.

AOX was found to be a convenient tracer of BKME: the effluents from the Weldwood of Canada mill at Hinton and the Procter and Gamble mill at Grande Prairie markedly increased AOX concentrations in the rivers. Downstream of the effluents AOX declined in concentration, primarily due to dilution by tributary inflow, but the total mass of AOX present in the water was fairly constant. During October 1989 about one third of the AOX mass disappeared from the water column along 240 km of the Wapiti-Smoky river system. During the winter surveys, there was no measurable loss of AOX from BKME over 240 km of the Wapiti-Smoky Rivers and over 1250 km of the Athabasca River. Supplementary sampling indicated that the AOX persisted even farther downstream, into the Peace and Slave Rivers.

The chlorinated organic material accounting for AOX in BKME is thought to be 80 - 90% chlorinated lignin. Chlorolignin is fairly soluble and fairly resistant to decay, which is consistent with the high persistence of AOX observed here. Chlorolignin is fairly non-toxic, however, some researchers report that chlorolignin can degrade slowly to more toxic chlorophenolics.

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1. INTRODUCTION

Adsorbable Organic Halide (AOX) refers to the amount of halide, principally chloride but also bromide and iodide, contained in organic compounds and recoverable during the analytical method. AOX has become an accepted measure of total chlorinated organic material, particularly that discharged by pulp mills using chlorine bleaching. Chlorinated organic material is of environmental concern because of the persistence and toxicity of some chlorinated organic compounds.

An AOX method has recently been implemented by the Alberta Environmental Centre, and is being used on bleached kraft pulp mill effluents (BKME) by Standards and Approvals Division (S&AD) of Alberta Environment to quantify chlorinated organic discharges. The Environmental Quality Monitoring Branch (EQMB) has initiated AOX sampling on rivers receiving BKME to assess the fate of such material. This report documents the results of receiving water sampling to date.

2. METHODS

Three surveys were carried out during which AOX was sampled:

Wapiti Smoky rivers	- October 1989
	- Feb-March 1990
Athabasca River	- Feb-March 1990

These surveys progressed downstream at approximately the same speed as the river's time-of-travel and generally employed the methods described by Noton and Shaw (1989). Effluents, tributaries, and the mainstem rivers were sampled, and supplementary samples were also collected from the Peace and Slave rivers (Figure 1). Procter and Gamble Cellulose Ltd. discharges BKME

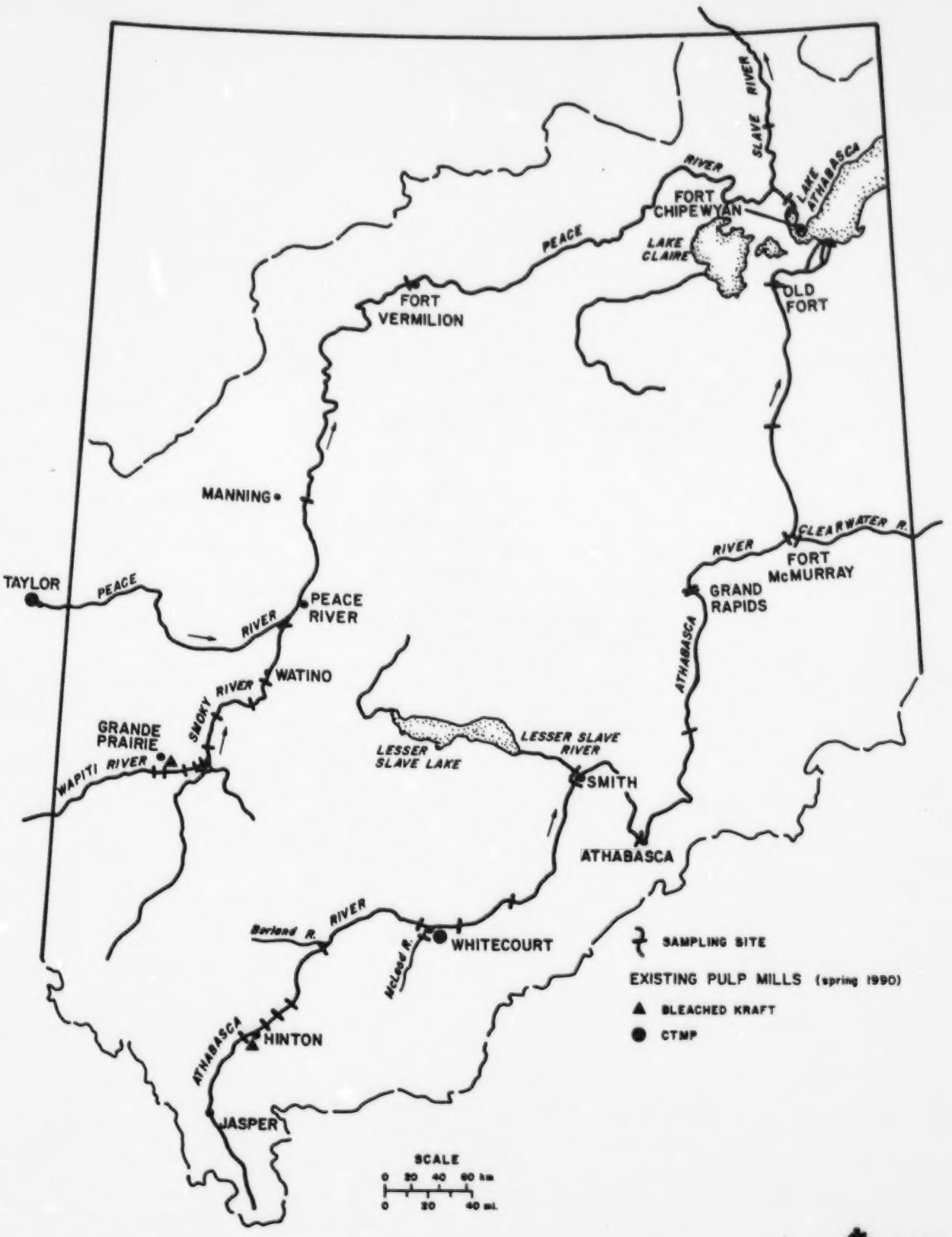

Figure 1. Sampling sites for AOX, 1989-90.

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near Grande Prairie and Weldwood of Canada Ltd. discharges BKME at Hinton. During both surveys, Procter and Gamble was employing about 25% ClO_2 substitution in its bleaching process to reduce chlorinated organic discharges and treating wastewaters in a 8-10d retention aerated lagoon. Weldwood was treating wastewaters in a 5-7d retention aerated lagoon and employing about 15% ClO_2 substitution. The pulp mills at Taylor, B.C. and Whitecourt do not use chlorine bleaching. In August, 1990, sampling again was conducted at some of the sites along the Athabasca River to obtain supplemental data.

Whole, unfiltered samples for AOX were collected in 500mL polyethylene bottles, acidified with nitric acid, and analyzed by the Alberta Environmental Centre, Research and Methods Development Branch. The analytical method (#E128.0; NAQUADAT #95080L) involves adsorption on activated carbon, combustion, and micro-coulometric titration, and is consistent with the Scandinavian Pulp, Paper and Board Testing Committee, Standard Method SCAN-W 9:89 (1989). The method is designed to measure both dissolved and particulate organic halogens. Trip (field) blanks were analyzed as per the method specifications. In addition, blind field blanks and blind duplicate samples were regularly submitted by the field crews. In August 1990, one sample from the Athabasca River was collected, mixed thoroughly in a stainless steel pail, split into eight replicates and analyzed to assess analytical precision.

Flows during the surveys were measured or estimated by Technical Services Division, Alberta Environment. Mass transport or 'load' of AOX was calculated as the product of concentration and flow, and expressed as kg/d, which is the unit used in effluent licencing: mg/L x m^3/s x 86.4 = kg/d.

3. RESULTS AND DISCUSSION

3.1 Quality Assurance

Field blank samples are checks on sample contamination from all potential sources (sampling, handling, laboratory), and AOX in such samples was low (0.000 to 0.004 mg/L, Table 1). Duplicate samples were submitted on 8 occasions and the percent difference within each sample pair was usually 0 - 4% (Table 1). Duplicate samples provide a check on all sources of variability that may affect the result. Analysis of the replicate split sample gave a standard deviation of 0.0014 mg/L at a concentration of 0.026 mg/L AOX. This is typical of the precision achieved with the method (I. Johnson, pers. comm.).

Flow measurements are also subject to errors, particularly during ice-covered conditions (G. Coles, pers. comm., Pelletier 1988) but replicate measurements to assess error are rarely done. However, since flow was measured sequentially downstream at approximately the rivers' time of travel, the flows can be summed to check their balance and provide an empirical assessment of accuracy. This is discussed below for each survey.

3.2 Wapiti and Smoky Rivers

During the synoptic survey of 2-5 October 1989, AOX was present at only trace concentrations in the upper Wapiti and upper Smoky rivers, but increased substantially in the Wapiti River downstream of the Procter and Gamble Cellulose Ltd. bleached kraft mill effluent (Table 2, Figure 2). The survey was carried out during near-average fall flows. Tributaries and other effluents contained insignificant loads of AOX. Mixing of the Wapiti with the Smoky diluted concentrations, but AOX was still present at the mouth of the

Table 1. Quality Control Samples for AOX

FIELD BLANK SAMPLES		DUPLICATE SAMPLES		
Date	AOX mg/L	Location & Date	AOX mg/L	% Difference from mean
		ATHABASCA SURVEYS		
14 Feb 90	0.000	AR u/s Hinton	0.000	0
8 Mar 90	0.002	14 Feb 90	0.000	
		Hinton Combined Effluent	3.3	3
		14 Feb 90	3.5	
		AR at Athabasca	0.030	9
		01 Mar 90	0.036	
		AR u/s of Horse R.	0.022	4
		14 Mar 90	0.024	
22 Aug 90	0.000	Hinton Combined Effluent	9.8	4
		21-22 Aug 90	10.7	
		WAPITI-SMOKY SURVEYS		
03 Oct 89	0.004	Procter & Gamble Effluent	23.1	2
		03 Oct 89	24.2	
06 Mar 90	0.001	Procter & Gamble Effluent	32	3
		27 Feb 90	33	
		Wapiti R. at Railroad Br.	1.3	0
		28 Feb 90	1.3	

REPLICATES

AR at Emerson Lakes Bridge - 22 Aug 90

AOX-mg/L	
0.027	n = 8
0.026	mean = 0.0260
0.024	std. dev. = 0.0014
0.024	coeff. var. = 5%
0.028	
0.027	
0.026	
0.026	

AR = Athabasca River

Table 2. Adsorbable Organic Halides (AOX) in the Wapiti, Smoky, and Peace River, 1989-90.

SITE	DISTANCE km d/s	OCTOBER 1989 DATE	FLOW m^3/s	AOX mg/L	AOX kg/d	FEB-MAR 1990 DATE	FLOW m^3/s	AOX mg/L	AOX kg/d
Wapiti R. @ Hwy 40	0	02 Oct	72.0	0.014	87	27 Feb	14.3	0.000	0
Grande Prairie STP Effluent	2	02 Oct	0.214	0.059	1.1	27 Feb	0.230	0.061	1.2
Wapiti R. d/s Haul Bridge	7.5	-	-	-	-	27 Feb	14.3	0.000	0
Procter & Gamble Storm Sewer/Cooling Water	9	03 Oct	(0.1)	0.052	0.4	28 Feb	0.048	0.000	0.02
Wapiti R. u/s of Mill Effluent	11	03 Oct	67.3	0.012	70	-	-	-	-
Procter & Gamble Final Effluent - ASB outflow	12	03 Oct	0.749	23.1	1490	27-28 Feb	0.516	32	1400
				24.2	1570	-	-	-	-
- Foam pond	12	-	-	-	-	28 Feb	0.516	33	1500
Wapiti R. @ Railroad Bridge	14	03 Oct	72.1	0.29	1800	28 Feb	14.5	1.3	1600
		-	-	-	-			1.3	1600
Wapiti R. u/s Bear R.	27	04 Oct	77.6	0.28	1900	28 Feb	14.5	1.4	1800
Bear R. mouth	27	04 Oct	2.23	0.034	6.5	-	-	-	-
Wapiti R. near mouth	44	04 Oct	80.6	0.27	1900	01 Mar	14.5	1.2	1500
Smoky R. u/s of Wapiti R.	43	04 Oct	106	0.004	40	01 Mar	24.7	0.003	6
Smoky R. @ Hwy 34	61	05 Oct	217	0.087	1600	06 Mar	61.1	0.34	1800
Smoky R. u/s of Puskwaskau R.	101	-	-	-	-	06 Mar	61.2	0.38	2000
Smoky R. @ km 105	142	-	-	-	-	07 Mar	64.8	0.34	1900
Smoky R. @ Watino	182	05 Oct	254	0.064	1400	07 Mar	81.0	0.31	2200
Smoky R. near mouth	246	05 Oct	279	0.056	1300	07 Mar	85.4	0.27	2000
Supplementary Sampling:									
Peace R. East of Manning	354	-	-	-	-	28 Feb	1061	0.016	1500
						27 Mar	1392	0.014	1700
Peace R. @ Ft. Vermilion	682	-	-	-	-	28 Mar	1294	0.030	3600

( ) Estimated values

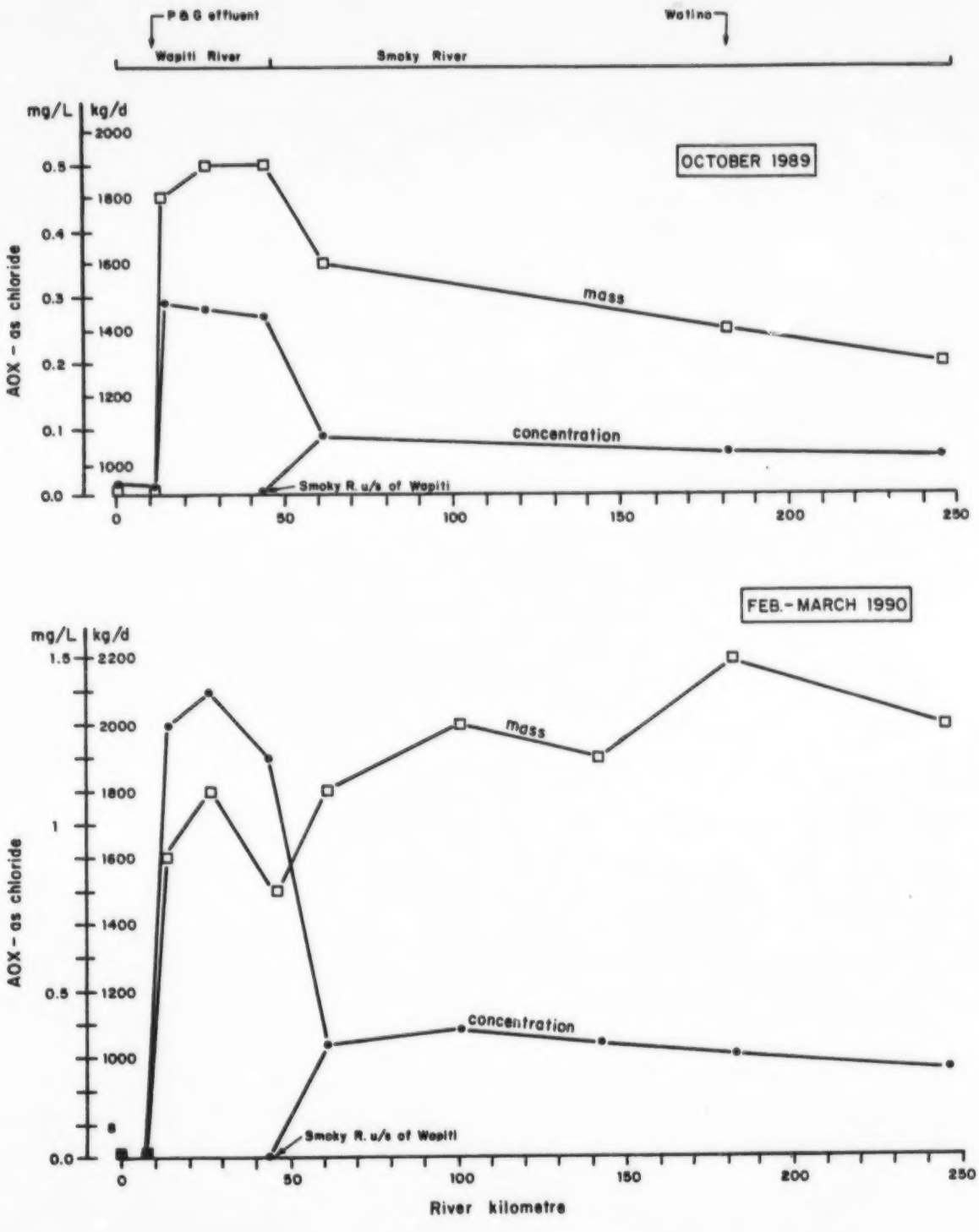

Figure 2. Adsorbable Organic Halides (AOX) in the Wapiti - Smoky Rivers, 1989 - 90.

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Smoky, some 240 km downstream of the mill. The load calculations indicate that about one third of the AOX mass in the Wapiti downstream of the mill had disappeared from the water column by the mouth of the Smoky. However, there may be error in the load estimates since they also indicated about 20% greater load in the Wapiti than in the BKME. Such error probably results primarily from inaccuracy in flow measurements and travel time estimates.

During the winter survey, AOX was again negligible in the Wapiti River upstream of the pulp mill, in tributaries, and in other effluents (Table 1). The bleached kraft mill discharged about 1450 kg/d of AOX which markedly increased AOX concentrations in the Wapiti River (Figure 2). Concentrations declined substantially upon mixing with the Smoky River, and then declined slightly down the length of the Smoky. However, the mass of AOX in the water column did not decline (Figure 2), indicating that concentration declined due to dilution by tributaries. The AOX mass increased about 25% down the river during the survey. There was significant meltwater inflow at the time and this may have increased the errors in flow measurement and travel time estimates, and/or contributed additional AOX.

Supplementary sampling downstream in the Peace River revealed AOX concentrations of about 0.015-0.030 mg/L (Table 2). This indicates that chlorinated organic material originating from the kraft mill on the Wapiti River was detectable downstream in the Peace River, however, further investigation would be required to confirm this since sampling was not done at the rivers' time-of-travel (thus continuity of mass cannot be assumed), and since the Peace River upstream of the Smoky River was not sampled.

3.3 Athabasca River

In February 1990, AOX in the Athabasca River upstream of Hinton was

not detectable, but increased to about 0.080 mg/L due to discharge of the Hinton Combined Effluent (HCE) (Table 3 and Figure 3). Concentration declined progressively downstream along the river, but AOX was still detectable at the river's mouth. The load of AOX in the water was negligible upstream of Hinton, but the HCE discharged about 230 kg/d to the river (Table 3). This mass remained in the water column throughout the length of the river (Figure 3). Note that the pulp mill at Hinton was shut down for part of February, 1990, and the AOX load in its effluent during this survey was lower than normal.

The load of AOX actually increased down the river, from about 230 kg/d near Hinton to about 300 kg/d in the lower reach of the Athabasca River. Other measured AOX inputs include (Table 3):

Other Effluents	5 kg/d
McLeod River	3
Lesser Slave River	26
Clearwater River	30
	64 kg/d

Unsampled tributaries accounted for about 107 m^3/s of flow during the survey. If it is assumed they contained AOX concentrations similar to the McLeod, Lesser Slave, and Clearwater Rivers (about 0.005 mg/L, Table 3), they would have contributed 46 kg/d, for a total of 110 kg/d of AOX input from sources other than the HCE. In view of the error to be expected in flow measurements, travel time estimates, and analytical procedures, this load of 110 kg/d likely accounts for the observed increase of 70 kg/d. Thus, about 75% of the AOX transported to Lake Athabasca during this survey appeared to

Table 3. Adsorbable Organic Halides (AOX) in the Athabasca River, Feb - March 1990

SITE	DISTANCE km d/s	DATE 1990	FLOW m^3/s	AOX mg/L	AOX kg/d
Athabasca R. u/s of Hinton	0	14 Feb	33.0	0.000 0.000	0 0
Hinton Combined Effluent (HCE)	1.9	15 Feb	0.784	3.3 3.5	220 240
Athabasca R. 6 km d/s of HCE	7.5	15 Feb	33.1	0.077	220
Athabasca R. 19 km d/s of HCE	23	15 Feb	33.4	0.089	260
Athabasca R. 36 km d/s of HCE	39	16 Feb	33.8	0.084	250
Athabasca R. u/s of Berland R.	104	16 Feb	36.0	0.074	230
Athabasca R. u/s of Whitecourt	210	20 Feb	50.7	0.047	210
McLeod R. @ mouth	211	20 Feb	10.1	0.003	3
Millar Western Effluent (CTMP mill)	212	20 Feb	0.136	0.12±	1.4
Whitecourt STP Effluent	215	20 Feb	0.039	0.11	0.37
Athabasca R. 10 km d/s McLeod R.	221	21 Feb	61.8	0.037	200
Athabasca R. @ Ft. Assiniboine	306	21 Feb	64.4	0.041	230
Athabasca R. u/s of Smith	438	28 Feb	73.3	0.047	300
Slave Lake STP Effluent		26 Feb	0.033	0.20	0.57
Lesser Slave R. mouth	449	01 Mar	43.7	0.007	26
Athabasca R. @ Athabasca	556	01 Mar	117	0.030 0.036	300 360
Athabasca STP Effluent	557	06 Mar	0.011	0.18	0.17
Athabasca R. near MacMillan Lake	678	06 Mar	123	0.026	280
Athabasca R. u/s Horse R.	946	14 Mar	142	0.022 0.024	270 290
Clearwater R. @ mouth	951	14 Mar	58.6	0.006	30
Ft. McMurray STP Effluent	951	14 Mar	0.180	0.16	2.5
Suncor Process Effluent	981	14 Mar	0.303	0.027	0.71
Athabasca R. near Bitumount	1029	15 Mar	207	0.019	340
Athabasca R. @ Old Fort	1157	21 Mar	218	0.014	260
Athabasca R. @ Lake Athabasca	1239	21 Mar	247	0.013	280
Supplementary Sampling:					
Lake Athabasca near Potato Island		21 Mar	-	0.020	
Riviere des Rochers		21 Mar	745	0.010	
Slave R. near laButte - LB		21 Mar		0.014	
- RB		21 Mar		0.016	
- mean			(2550)	0.015	

( ) Estimated value

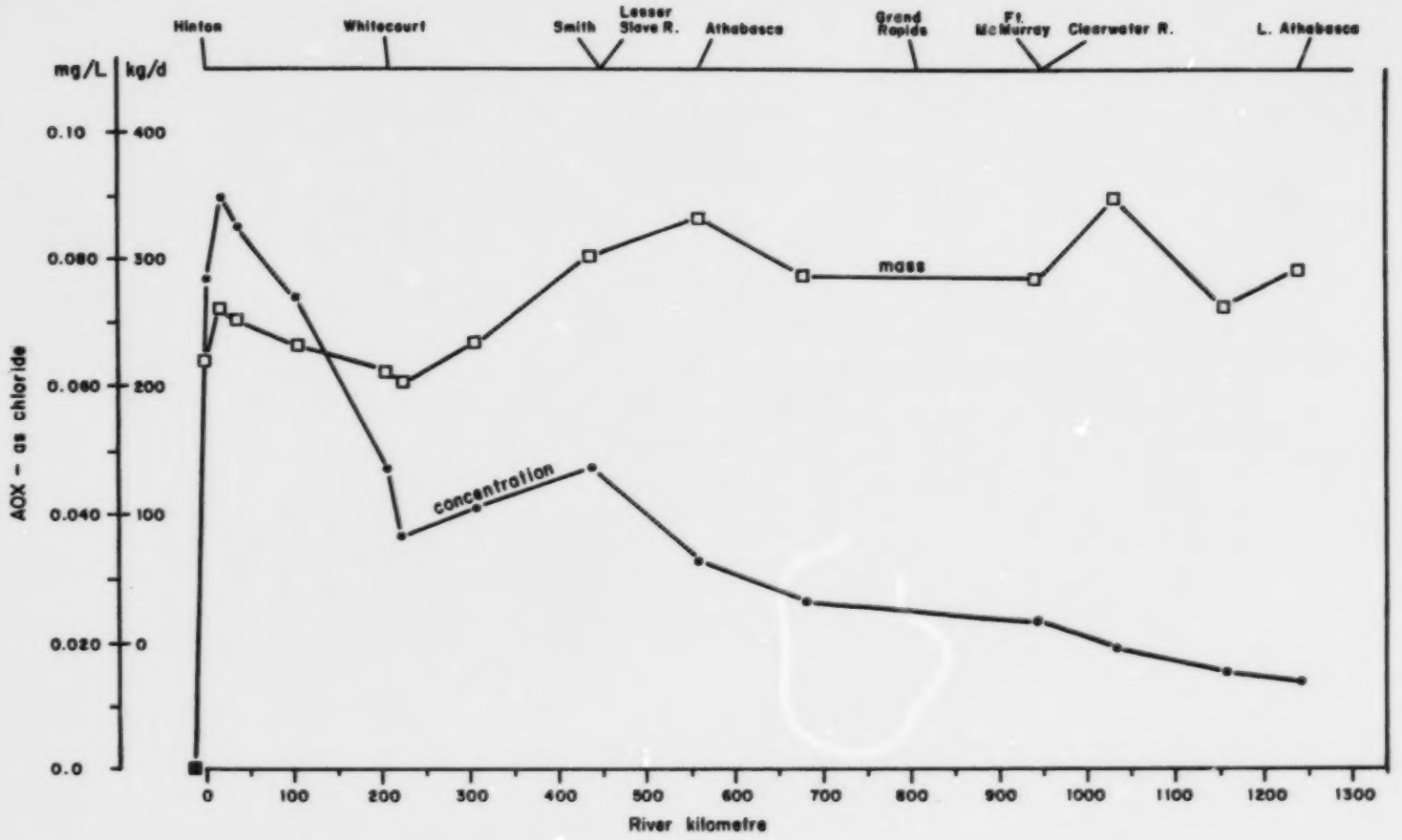

Figure 3. Adsorbable Organic Halides (AOX) in the Athabasca River, Feb. - March 1990.

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originate from the Hinton combined effluent. AOX from the HCE showed no measurable disappearance from the water column over that 1250 km distance.

Supplementary sampling farther downstream in Lake Athabasca and the Slave River (Table 3, Figure 1) revealed AOX concentrations similar to those in the lower Athabasca River (0.010 - 0.020 mg/L). This suggests the AOX persists farther downstream, however, more sampling would be required to confirm this since background levels of AOX in unaffected water bodies in that area are not known.

3.4 General Discussion

3.4.1 Nature of AOX

The identity of the organochlorine compounds from bleached kraft pulp mills effluents has been investigated by a number of researchers. It has been estimated that 80 - 90% of the organically bound chlorine (AOX) is in high molecular weight compounds (>1000 MW), generally referred to as chlorolignins (Salkinoja-Salonen et al, 1981; Kringstad and Lindstrom, 1984; Voss 1987). This material is fairly soluble and once discharged it appears to remain in the water column, as indicated by the findings here, and can serve as a tracer of BKME. AOX has been used as a tracer of BKME from Swedish mills on the Bothnian Sea (Sodergren 1989), although the researchers note that AOX can originate from sources other than BKME and therefore must be used as a tracer with caution. AOX from BKME will also include lower molecular weight compounds, some of which, are of environmental concern (e.g. chlorophenolics).

3.4.2 Background Conditions

The presence of measurable amounts of AOX at sites not influenced by

BKME or municipal effluent suggests that there are naturally occurring organic compounds containing halogen atoms in surface waters. This has been reported elsewhere and the AOX concentration has been linked to the concentration of total organic carbon (TOC), and humic and fulvic acids (Wigilius et al 1988; Paasivirta et al 1988; Grimvall 1990). Background sites in this survey had the following AOX and TOC concentrations:

		AOX (mg/L)	TOC (mg/L)	AOX:TOC (mg/g)
Athabasca R. u/s Hinton	est.	0.0005	0.64	0.8
*McLeod R. mouth		0.003	4.43	0.7
*Lesser Slave R. mouth		0.007	11.0	0.6
Clearwater R. mouth		0.006	6.36	0.9
Wapiti R. u/s Grande Prairie	- Oct 89	0.014	6.57	2.1
	- Mar 90 est.	0.0005	2.57	0.2
Smoky R. u/s Wapiti R.	- Oct 89	0.004	5.9	0.7
	- Mar 90	0.003	1.6	1.9

*Receive Municipal sewage, but AOX apparently not affected.

The values are near the lower limit of detection, and are at the low end of background values reported by Wigilius et al (1988) of 0.010 - 0.050 mg/L and by Martinsen et al (1988) of 0.010 - 0.015 mg/L. The mean AOX:TOC ratio of about 1 mg/g is also at the low end of background values reported by Wigilius et al (1988) and Grimvall (1990) of 1 - 5 mg/g and 0.7 - 8.6 mg/g, respectively.

3.4.3 Persistence

The findings here indicate no measurable loss of AOX from BKME in the two receiving river systems during winter, over the length of river investigated. The distance from Hinton to Lake Athabasca is about 1250 km and the travel time was about 35 days. During the fall survey, there was an

indication of some disappearance of AOX over the 240 km of the Wapiti-Smoky river system (travel time about 4 - 5 days), however, further investigation would be necessary to confirm the loss. The downstream persistence of AOX in winter in the Wapiti-Smoky mirrors the persistence reported there for organic carbon, colour, and phenolics from the BKME (Noton et al 1989). Conditions during the winter surveys included water temperatures of 0°C, ice cover, and low suspended solids (1 - 5 mg/L), whereas during the fall survey water temperatures were about 7 - 9°C and suspended solids about 20 - 45 mg/L. Differences in these and other variables may be related to the apparent downstream loss of AOX in the fall.

No studies of AOX persistence in receiving rivers could be found in the scientific literature, although persistence of AOX in the Gulf of Bothnia (Sodergren 1989) and of other pulp mill effluent constituents in receiving waters (e.g. Brownlee and Strachan 1976; Merriman 1988) have been reported. Chlorophenols attributable to bleached kraft pulp mills on the upper Fraser River have been detected in the Fraser River Estuary, some 500 km downstream (Carey and Hart, 1988). A March 1990 sampling of the lower Athabasca River, downstream of Ft. McMurray, revealed chlorophenols to be present in the ng/L range (B. Brownlee, pers. comm.). The probable source of these compounds is the bleached kraft mill at Hinton, about 1000 km upstream.

3.4.4 Environmental Significance

Although AOX standards for pulp mill effluents are being set in several jurisdictions, no surface water quality objectives exist for the sum parameter AOX. Most of the AOX from BKME is probably chlorolignins, which because of their high molecular weight cannot penetrate cell membranes and

exert toxicity (Salkinoja-Salonen et al 1981; Kringstad and Lindstrom 1984, Sagfors and Starck 1988). However, chlorolignin imparts colour to water which may have aesthetic impact and which may reduce light penetration and photosynthesis. As well, chlorolignin has been demonstrated to undergo biochemical breakdown under environmentally relevant conditions to more toxic chlorophenolic compounds, albeit at relatively slow rates (Erikson and Kolar 1985; Erikson et al 1985). Even though downstream disappearance of AOX was not observed in the winter surveys, such breakdown or biotransformation may be occurring: the resolution of the AOX measurement (µg/L) may be too gross to detect such changes (ng/L) and/or the breakdown products may also remain in the water column and be measured as AOX (e.g. soluble chlorophenolics).

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